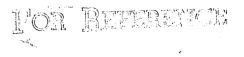
NASA TECHNICAL MEMORANDUM

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AEROSIL (R) FOR THE IMPROVEMENT OF THE FLOW BEHAVIOR OF POWDERED SUBSTANCES



IMENAN MANAY MOMPA RADIAN AR OF FOIL

Translation of "AEROSIL (R) zur Verbesserung des flierenREIHE PIG-Bverhaltens Pulverformiger Substanzen Degussa", Frankfurt, MENIE West Germany, No. 31, August 10, 1971, pp. 1-17.

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The highly dispersed silicic acid AEROSIL (R) has been used for many applications in numerous branches of industry. In the course of developing new methods of producing silicic acids, new products have been introduced with unusual properties, especially for application in powdered materials.

Powdered substances tend to agglomerate and stick together whatever their composition and use. This impedes the packing, storage and handling of this material. However, increasing simplification in industry demands good flow behavior of these powdered materials as well as stability in storage, weathering and under pressure.

## 1. Survey of the literature

The attempts to improve the flow properties of powdered products are mirrored in a multiplicity of patents and treatises. However, these are mostly limited to the description of individual cases.

In this way the following products are named, for example, as "flow facilitators" or "flow helpers" for ammonium fertilizers. alipathic amine in combination with carrier materials hydrazine and guanidine compounds urea-formaldehyde resin solutions phthalocynanine compounds sprays of high molecular weight glycols paraffin sulfo-phenolic acid and alkyl-aryl-sulfonate fatty acid aluminum salts

 $<sup>^{\</sup>star}$ Numbers in margin indicate pagination of foreign text

chromium, cadmium and tin salts, also such simple organic acids including iron ammonium citrate alkali- and alkaline earth phosphate magnesium oxide and calcinated dolomite hexacyanoferrate copper-I-oxide, copper-II-oxide, also zirconium-oxychloride boron trioxide rice flour

This enumeration -- only a selection from a far greater number of publications -- shows the multiplicity of the recommended flow agents. These have been applied in different forms, such as: mixtures, sprays and coatings. Soon sylicic acids and silicates found a more uniform and, therefore, more clearly arranged application. Of these, the type obtained from the gas phase, e.g., the AEROSIL types have especially found increasing use by manufacturers and processors of powdered pro-Some examples from the literature are: prevention of clumping of fertilizers (1) improved flow of pickling salts (2) flow behavior of foods, drugs and cosmetics (3) grinding and free-flowing aid for butyl-hydroxyl-toluol, methionine and its calcium salts, sodium proprinate, urea, vitamins, diet products, spices, etc. (4) flow agent for sodium chlorate and sodium chloride (5) for the loosening of malt and molasses preparations (6) for improving the flow of powdered waves (e.g., Carbowachs 600) (7)

## 2. Basics of flow behavior of powders and the mode of action of flow agents

The flow behavior of powders is determined principally by the physical properties of the powder: hygroscopicity or sensitivity to weathering form and size distribution of the powder particles

ability to become electrostaticallay charged sintering at low temperatures adhesion of the particles to each other

Investigations of the influence of these factors and suggestions for improving of flow behavior are displaced in the newer literature in the following examples:

- 2.1. M. Frey (8) indicates that for crystalline products even traces of moisture produce an extremely thin film of fluid, often in the form of a saturated solution of the product. This acts like a binding agent in causing the particles to adhere and thus hinders their "free flowing".
- 2.2. R. Tawashi (9) reports about the influence of friction between the particles as a quantity obstructing the flow behavior. He also mentions the possibility of forming a "single particle" covering of the powder particles by means of the addition of small amounts of highly dispersed flow agents, e.g., sylicic acids. Such a covering of the powder particles by a single layer hull of flow agent would allow a minimal value to be achieved for the friction between different particles. The optimal amount of flow agent can be calculated as a function of the density and average particle size (10).
- 2.3. <u>F. Gstirner and C. Pick</u> (11) refer to the difference between specific heavy powdered material with good flow properties and specific light powder materials which mostly flow poorly. For the latter the ability to flow can be improved by means of highly dispersed flow agents from 2 to 8 times.
- 2.4. <u>W. A. Ritschel</u> (12) describes the electrostatic charging of powders as a cause of disturbance in their flow

behavior and of the appearance of reagglomeration on grinding of such materials. He specifies the addition of highly dispersed sylicic acid as a remedy for this.

2.5. <u>H. von Czetsch-Lindenwald and A. F. Asker</u> (13) reports about the use of flow agents in improving the measurement of dosage in medicinal materials for the correct filling of capsules. Here, too, sylicic acids are recommended as an effective flow agent.

## 3. AEROSIL as a flow agent

The following demands for an effective flow agent with the most general possible application result from investigations of the theory of flow behavior.

- 3.1 Extreme particle fineness for the single layer covering of the material used with only small additions. Figure 1 shows the particle fineness of AEROSIL 200 by means of an electron micrograph.
- 3.2 The loosest possible agglomeration of the primary particles which largely also divides into the actual effective primary particle form or minimal agglomeration through simple mixing procedures.
- 3.3 Largely inactive chemically with the materials to be used.
- 3.4 High chemical purity and no physiological problems for application for pharmaceuticals, human and animal food.
- 3.5. Absolute colorlessness, so that no discoloration of the substances to be used need be feared.

3.6 Hydrophilic or hydrophobic surface properties choosable for specific cases.

The demands named under 3.1 and 3.6 have been fulfilled by the highly dispersed sylicic acids and aluminum oxide produced according to the AEROSIL procedures (14). In the past years AEROSIL has increasingly been used as a grinding and flow agent. It is produced from silicon tetrachloride by flame hydrolysis and is the purest form of sylicic acid produced large scale (15).

In addition to a high degree of chemical purity, hygienic harmlessness, high loose volume and extreme particle fineness are outstanding properties of AEROSIL for its use as a flow agent. The particle size distribution of AEROSIL 200 is shown in Figure 2. As a consequence of its high absorbtive capacity for fluids of all types, AEROSIL is appropriate for the conversion of liquid, plastic lubricating or agglomerated mixtures of materials to "dry" largely pressure stable and free flowing powdered products. Of the different kinds of AEROSIL, AEROSIL 200 is recommended for this application.

AEROSIL (R) 972 is a special type AEROSIL and differs principally in its surface properties. While AEROSIL 200, like all the usual sylicic acids, is hydrophilic, AEROSIL R 972 is hydrophobic. This is brought about by replacing the sinanol groups on its surface with di-methyl-dichlorosilane (16).

A consequence of the hydrophobic character of AEROSIL R 972 is its ombrophobicness in water. In many cases, this is of decisive significance for its outstanding effect as a flow agent in comparison with ordinary hydrophilic sylicic acids. Its use as a flow agent represent considerable progress for the methods of improving the flow behavior of powdered materials.

Analogously to the AEROSIL procedure, aluminum oxide C is also produced from aluminum chloride by flame hydrolysis and, according to current experience, is principally useful in reducing the tendency of powdered materials to acquire electrostatic charge.

### 4. Mixing technique

The special properties of AEROSIL 200 and AEROSIL R 972 as flow agents (especially AEROSIL R 972), namely, extreme particle fineness and loose secondary structure, can also be optimally effective if the method of mixing leads to a good coating of the powdered particles by the AEROSIL.

According to extensive investigations, the following method of mixing is the most effective:

All the AEROSIL is placed in a mixer which operates according to the free fall principle, and the powdered product to be coated is added in approximately 4 or 5 portions under continuous mixing. The distribution of the AEROSIL then succeeds according to the model of the origin of a "snowball" and can be determined in the following example both mathematically and experimentally.

For an addition of 1% AEROSIL with an average effective secondary particle size of 0.5 microns (taken as an example) to sodium hydrogen carbonate, more than 1000 secondary particles of AEROSIL are available for coating each sodium hydrogen carbonate particle. In this only the experimentally useful secondary structure of the AEROSIL is employed and not its primary particle sizes since here more than 10 million AEROSIL

primary particles would be available for coating each sodium hydrogen carbonate particle.

The degree of agreement between this mathematical example and practice is shown in the following experiment in which hydrophobic AEROSIL R 972 is used in coating the particles for better visibility:

Procedure: while the untreated sodium hydrogen carbonate is quickly wet by water and sinks, the product which was treated with 1% AEROSIL R 972 remains persistently on the surface of the water without becoming wet (see Figure 3).

This method of mixing according to the "snowball principle" is based on the following processes:

With the addition of the first fifth of the powder being treated into the entire amount of the AEROSIL applied, "snow-balls" form a loose, at first still surplus sheet of "snow" (AEROSIL) around the unequally large particles of the powder. A surplus of AEROSIL also remains. With the addition of the next fifth, the newly added powder particles take the AEROSIL necessary for coating them from the still available surplus AEROSIL. In the final state of the mixture, which should approach the ideal state as nearly as possible, the powder particles finally have approximately "mono particle" coatings of AEROSIL (see Figures 4 and 5, compare 2.2).

Had the method of mixing been reversed, the small amount of AEROSIL (0.5 to 1%) would have been coated or "swallowed" by the powder and the powder would have scarcely changed its flow behavior. In addition to the method of mixing, the process of mixing requires some attention: the powder component should be loosely mixed without too much pressure or shearing stress.

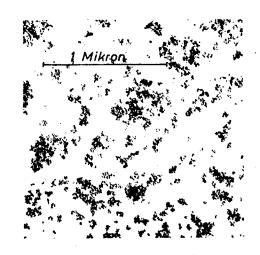


Figure 1. Electronmicrograph of AEROSIL 200

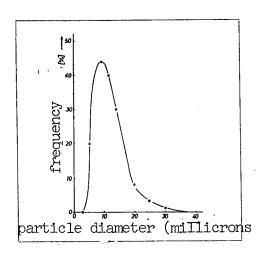


Figure 2. Particle size distribution of AEROSIL 200



Figure 3. Left test: NaHCO untreated; right test: NaHCO treated with 1% AEROSIL R 9723

For carrying out this type of mixing experiment, we found the "turbula" mixing for example to be quite useful. It was also sufficiently effective in producing so called "dry water" (see Figures 6 to 8)



Figure 4. NaHCO3 (without AEROSIL R 972)

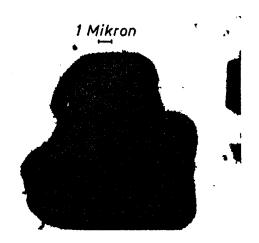


Figure 5. NaHCO $_3$  + 0.5% AEROSIL R 972

#### 5. Methods of measurement

The evaluation of a powder succeeds according to several points of view of which the following tests are the most important for practical concerns:

#### 5.1 Ability to flow



Figure 6. "Turbula" mixer Manufacturer: W. A. Bachofen, Basel (Switzerland)



Figure 7. Left: before mixing; right: after mixing

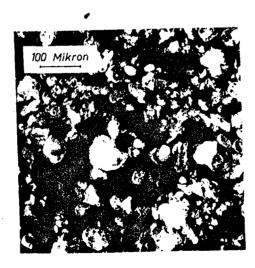


Figure 8. "dry water" consisting of 90% water and 10% AEROSIL R 972

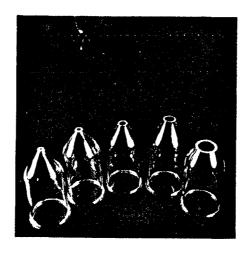


Figure 9. Measuring vessels

- 5.2 Dispersion
- 5.3 Resistance to pressure
- 5.4 Static charge

## 5.1 Ability to flow

Several methods have been proposed for measuring the ability of a powder to flow. These are based on running the powder through standardized funnels, measurement of the angle of inclination or elevation of the powder which runs out, or on the determination of the frictional resistance of the particles to each other.

Because the measurement of the "ability to flow" is ultimately concerned with "flowing" the simple method of viscosity measurement for fluids obviously suggested itself for the measurement of the flow of powders. This method employs vessels with different sizes of mouths to measure the viscosity.

For this purpose hour glass shaped glass vessels with different sized mouths were suggested and were chosen (see Figures 9 and 10). The vessels were made hydrophobic by rinsing

them with a silicon oil solution and leaving them in a 300°C drying cupboard for 6 hours.

#### 5.2 Dispersion behavior

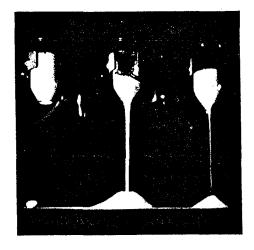
Apart from an ability to flow, a powder must have a good dispersion behavior for a number of applications. This is particularly true for all powdered materials which are sprayed or scattered (cosmetic powder, pest control agents, etc.). Because these materials are so finely divided and have such a low loose weight, their flow behavior can only be improved in unusual cases. This is also not at all necessary for this application assuming the dispersion behavior to be sufficiently good.

However, because dispersion behavior is very strongly influenced by humidity (storage, transport), the following weathering test was chosen to examine it:

The powder to be tested was weathered in an open weighing bottle under 65 or 80% relative humidity at 30°C. Its external behavior was periodically checked. (Insofar as it is of interest, the flow behavior can be tested at the same time, using the method described in 5.1) (Figures 11 to 14).

## 5.3 Resistance to pressure

Powdered products tend to agglomerate when stored in bags. The following test has proved useful in judging their "storage resistance"; the powder to be tested is placed in a cylinder of about 50 mm inner diameter to a height of about 20 mm. A 1.2 kg piston carrying an additional 2 kg of weight is placed on the powder. The duration of the pressure test can be chosen in accordance with the requirements of the powder with respect to packing and storage. From our own experience 24 hours under



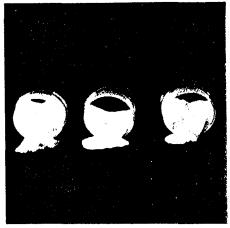


Figure 10. Example of the evaluation of flow behavior Left: NH<sub>4</sub>Cl without addition Note: 6 middle: NH<sub>4</sub>Cl with 0.5% AEROSIL R 972 note: 3; right: NH<sub>4</sub>Cl with 0.5% AEROSIL R 972 note: 1; (the example of "note 3" merely serves to demonstrate this note "3").

Figure 12. Before weathering Left: NaHCO4 (without addition) note 4; middle: NaHCO4 (+0.5% AEROSIL R 972) note 1; right: NaHCO4 (+1% Ca-stearate) note 1

pressure was sufficient for a judgement to be made. The pressure amounted to 0.17 kg/cm<sup>2</sup> with this test arrangement. This corresponded to the weight of 10 to 12 50 kg bags placed on top of each other. After 24 hours, the 2 kg weights were removed and the hollow cylinder was carefully raised. The powder remaining under the piston was then determined. (Figures 15 and 16).

## 5.4 Static charge

The static charge on a powder also considerably hinders its ability to flow. W. A. Ritschel (12) reported about this in detail.

A test which is simple to carry out is the energetic rubbing of the powder against the wall of a beaker with a glass or hard rubber rod.

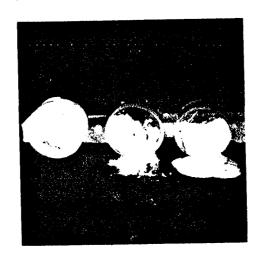


Figure 11. Example of a weather series. Left: evaluation note 6 (NH<sub>4</sub>Cl without R 972) after 2 days weathering; middle: evaluation note 5 (NH<sub>4</sub>Cl without R 972) unweathered; right: evaluation note 1 (NH<sub>4</sub>Cl + 1% R 972) after 6 days weathering

(for Figures 10-12) Weathering of sodium bicarbonate with and without the addition of AEROSIL R 972 or calcium stearate as a flow agent (weathering: 80% realtive humidity at 30°C).

In this way, powders which become charged form a stubborn coating on the beaker wall and glass rod. Uncharged powders, on the other hand, show no coating (Figures 17 and 18).

The disturbance of the flow behavior of spin sintered powders by accumulation of static charge is shown in a demonstration experiment (Figure 19).

W. A. Ritschel (12) shows the influence of static charge on the grinding of a powder for the example of proxyphyllin (obstruction of milling)\* (Figures 20 and 21).

<sup>\*</sup>With the friendly permission of Prof. Dr. W. A. Ritschel, Saeckingen, for which we thank him here.

TABLE 1.

Vessel No.

Evaluation No.	north diameter (mm)	evaluation for smooth running through of powder
1	2.5	very good (seldom used with
2	5	good powders)
3	7	good on the whole
4	12	adequate
5	18	deficient
6		substandard (did not pass through number 5)

All "hour glasses" had a height of 42 mm and an inner diameter of 90 mm

TABLE	· 2.

characteristic behavior
completely unchanged and flowing excellently (smoothly flowing through hour glass #2)
not adhering to the surface; practically unchanged from its pre-test state
e slight surface adherence; easily returned to the original dispersion
adhering to the surface; returns to the original dispersion by rolling and shaking
adheres; the original dispersion cannot be reached by shaking
agglomerated into clumps and moist to the touch





Figure 13. After 2 days weathering

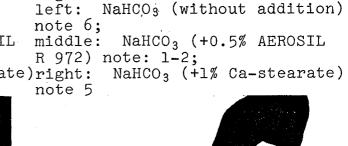
Left: NAHCO3 (without addition) note 5;

middle: NaHCO3 (+0.5% AEROSIL

R 972) note 1;

NaHCO3 (+1% Ca-stearate)right: NaHCO3 (+1% Ca-stearate) right:

note 4



ing



Figure 15. Test arrangement for resistance to pressure

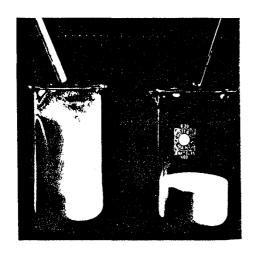


Figure 16. Pressure resistance of ammonium chloride

Figure 14. After 4 days weather-

left: ammonium chloride without addition

right: ammonium chloride with 1% AEROSIL R 972

Figure 17. Left: PVC without addition,

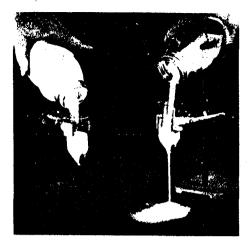
right: PVC + 1% aluminum oxide C

#### TABLE 3.

evaluation note	characteristic behavior
1 = very good	completely unchanged and flowing smoothly through hour glass #2
2 = good	partially loosely adhering; easily returned to the original state
3 = good on the whole	e loosely formed; can be returned to powder with slight finger pressure
4 = adequate	loosely agglomerated; still crumbles under finger pressure
5 = deficient	semi-firmly agglomerated; no longer crumbles under finger pressure
6 = substandard	solidly formed

# 6. Examples of the use of AEROSIL in improving the properties of powdered materials

- 6.1 Improvement of flow behavior (Figures 22 and 26)
- 6.2 Improvement of weathering stability of moisture sensitive material (Figures 23 and 24)
- 6.3 Improvement of pressure stability (stacking resistance (Figure 25)
- 6.4 Improvement of pressure sensitive mixtures of materials with regard to agglomeration and clumping (Figures 27 and 28)
- 6.5 Reduced sintering of powdered or granulated substances (Figures 29 and 30)
- 6.6 Loosening and disagglomeration of spray powders (Figures 32 and 33)
  - 6.7 Conversion of fluid materials to powedered form (Fig 31)
  - 6.8 Increase in volume of powdered materials (Figs 34 and 35)



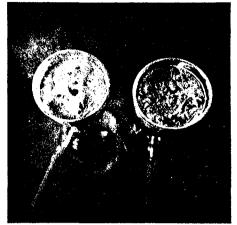


Figure 18. Flow behavior of statically charged and uncharged PVC powder.

Left: PVC powder without addition; right: PVC powder + 1% aluminum oxide C (both samples charged in the "Turbula mixer")

Figure 19. Left: PVC spin sintered powder without addition (powder shows hesitant flow and blowing out of the pressurized air through crater forming channels) right: PVC spin sintered powder with 0.2% AEROSIL R 972 and aluminum oxide C (powder flows homogeneously without blowing out of the powder. No air pressure channels!)

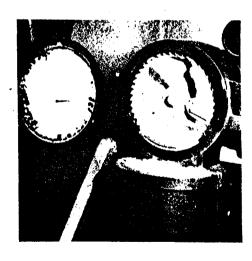


Figure 20. Grinding of proxyphyllin without addition

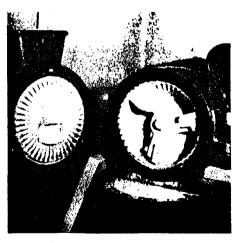


Figure 21. Grinding of proxyphyllin, to which 0.45% AEROSIL 200 was added

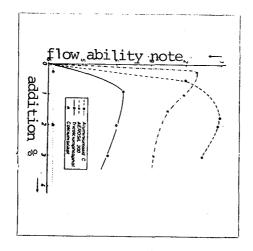


Figure 22. Effect of different flow agents on ground sulfur.

Flow agent 1: aluminum oxide C Flow agent 2: AEROSIL 200

Flow agent 3: tricalcium phosphate Flow agent 4: calcium silicate

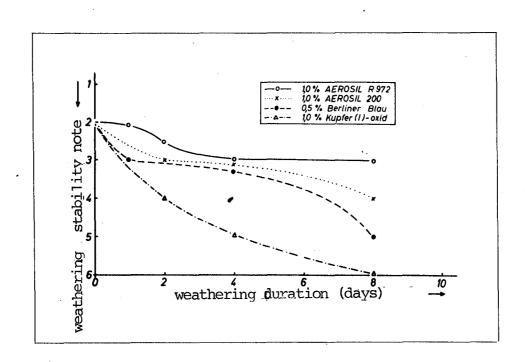


Figure 23. Weathering progress of ammonium nitrate with the addition of different flow agents (65% relative humidity, 30°C).

Flow agent 1: 1% AEROSIL R 972 (mixture not colored) Flow agent 2: 1% AEROSIL 200 (mixture not colored)

0.5% Berlin blue (mixture colored blue) Flow agent 3:

Flow agent 4: 1% copper-I-oxide (mixture colored red)

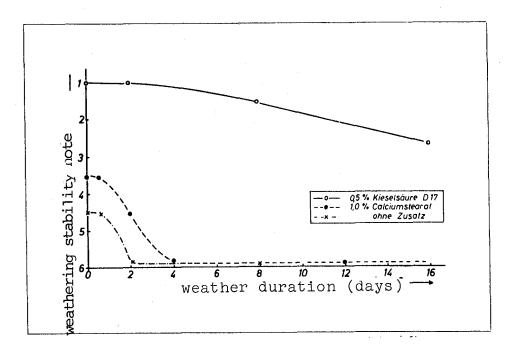


Figure 24. Weathering progress of sodium hydrogen carbonate with and without flow agent addition (65% relative humidity, 30°C)

Flow agent 1: 0.5% sylicic acid D 17 Flow agent: 1.0% calcium stearate Curve 3: without flow agent addition

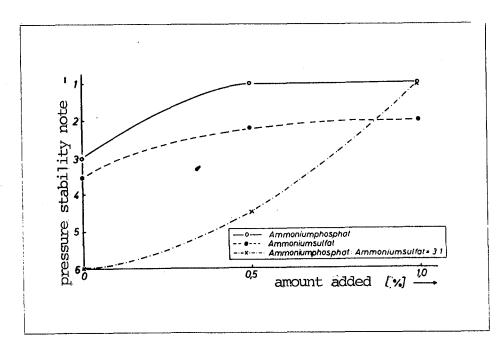


Figure 25. Pressure resistance with increasing addition of hydrophobic sylicic acid D 17

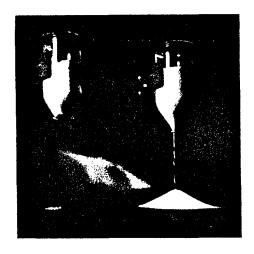
Curve 1: ammonium phosphate
Curve 2: ammonium sulfate

Curve 3: mixture ammonium phosphate: ammonium sulfate = 3.1

/12

/11

Figure 25: Remark: Example for the open observed phenomenon that the pressure resistance of material mixtures is worse than that of the individual components.



0.2% sylicic acid D 17

Figure 26. Left: sulfur with- Figure 27. Concentrated feed, out addition, right: sulfur + left: without addition, 0.2% sylicic acid D 17 right: with 10% AEROSIL 200

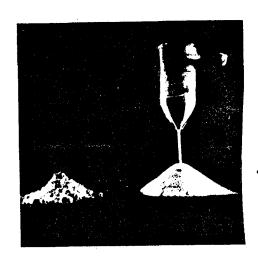


Figure 28. Powdered milk, left: without addition, right: with 0.5% sylicic acid D 17

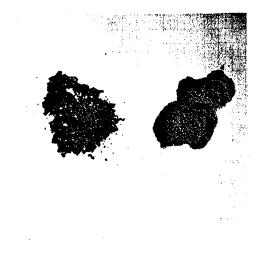
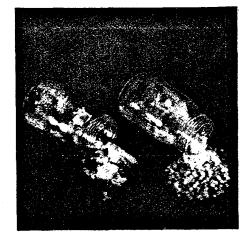


Figure 29. Artificial resin formed from phenol-formaldehyde, left: without addition (sample reduced for picture) right: with 1.5% AEROSIL 200 (both samples stored 7 years)



1 Mikron

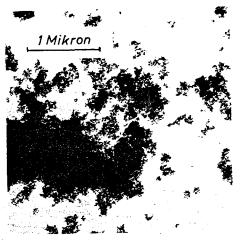
added AEROSIL 200

Figure 30. Paraffin geperite Left: without addition right: treated with 2% AEROSIL (both samples store 5 years)

Figure 32. Finely ground magnesium oxide (in the normal supply state)

Figure 34. Increase in loose volume of a body poweder with the addition of increasing amounts of AEROSIL 200





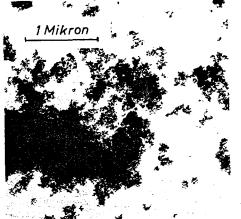


Figure 31. Conversion of 70% choline chloride solution to powdered form left: 70% choline chloride D 17. right: with 40% AEROSIL

Figure 33. Similar magnesium oxide mixed with 5% hydrophobic sylicic acid

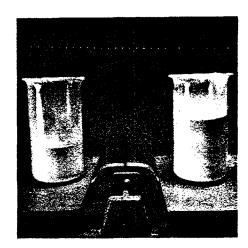


Figure 35. Equal weights of powder

left: without AEROSIL

right: with 10% AEROSIL 200

#### 7. Summary and prospects

The use of AEROSIL 200, AEROSIL R 972, aluminum oxide C and sylicic acid D 17 as flow agents has made broad improvements possible in the properties of powdered products.

In addition to flow and dispersion behavior, the sifting and spray behavior and the grinding and air separation
(also sifting) have been substantially improved. This is particularly true for the hydrophobic type AEROSIL R 972 which,
in addition to providing outstanding weathering stability,
can be used in certain cases to regulate the moisture absorbancy as desired.

Powdered materials containing AEROSIL as a flow agent can more easily and quickly be mixed to homogeneity and are far less subject to the danger of a subsequent separation (paints, artificial resins, etc.).

Sprayed materials and powders function more reliably with AEROSIL. Their improved dispersion behavior allows a substantial reduction in the cost of their application because the same amount of surface can be covered with smaller amounts of treated powder than is the case with untreated powder.

The conversion of fluid or plastic materials into powdered form by means of AEROSIL makes new forms of packing, handling and application possible. The high absorbability of AEROSIL by fluids and pastes is of particular significance in maximizing the concentration of the material in the product.

The often desensitizing effect of AEROSIL or spontaneously reactive materials and material mixtures (e.g., organic peroxides) makes a reduction in the dangers of handling and transport of these materials possible.

#### 8. LITERATURE

- (1) Prevention of caking of fertilizers, Patent 950 559 11.10 1956 (BASF)
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## PHYSICO-CHEMICAL DATA

<b>;</b>		AEROSIL 200	SYLICIC ACID D 17	AEROSIL R 972	ALUMINUM OXIDE C
appearance	•	loose white powder			
moisture absorbancy surface according to BET	m²/g	hydro- philic 200±25	hydro- philic 110±30	hydro- philic 120±30	hydro- philic 100±15
average size of the primary particles	milli- micron	12	18	16	10
compressed volumes (DIN 53 194)	M1/100g	1700	1650	2000	1600
drying losses (DIN 53 198 method A) (2 hrs at 105C)	96	<1.5	3	<0.5	<5
heat loss *) (DIN 52911) (2 hrs at 1000C)	8	<1	7	<2	<3
pH-value (DIN 53200) (in 4% aqeous emulsion)		3,6-4,3	6.5**)	3.5-4.1**	) · 4 <b>-</b> -5
SiO <sub>2</sub> *)	9	>99.8	92	>98.3	<0.1
Al <sub>2</sub> 0 <sub>3</sub> *)	ş	<0.05	<0.1	<b>~&lt;0.05</b>	>97
$Fe_20_3$	ક્ર	<0.003	<0.04	<0.01	<0.2
TiO <sub>2</sub>	8	<0.03	-	<0.03	<0.1
нст	<b>%</b> .	<0.025	<0.2	<0.05	<0.05
grit according to Mocker (DIN 53 580)	<b>&amp;</b> (:	<0.05	<0.05	<del>-</del>	

<sup>\*)</sup> relative to the substance which was dried for 2 hrs at 105C.

\*\*) in water: methanol 1:1

(table continued) ....

AEROSIL 200: Packing:

cylicic acid D 17:

paper bags to 10 kg net weight paper bags to 25 kg net weight

AEROSIL R 972 and

aluminum oxide C

paper bags to 5 kg net weight

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